INTERNATIONAL STANDARD 1055

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Zinc and zinc alloys — Determination of iron content — Spectrophotometric method

Zinc et alliages de zinc — Dosage du fer — Méthode spectrophotométrique

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO Member Bodies). The work of developing International Standards is carried out through ISO Technical Committees. Every Member Body interested in a subject for which a Technical Committee has been set up has the right to be represented on that Committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

Draft International Standards adopted by the Technical Committees are circulated to the Member Bodies for approval before their acceptance as International Standards by the ISO Council.

Prior to 1972, the results of the work of the Technical Committees were published as ISO Recommendations; these documents are now in the process of being transformed into International Standards. As part of this process, Technical Committee ISO/TC 18 has reviewed ISO Recommendation R 1055 and found it technically suitable for transformation. International Standard ISO 1055 therefore replaces ISO Recommendation R 1055-1969 to which it is technically identical.

ISO Recommendation R 1055 was approved by the Member Bodies of the following countries :

Australia	India	Poland
Belgium	Iran	South Africa, Rep. of
Brazil	Ireland	Spain
Canada	Israel	Sweden
Chile	Italy	Turkey
Czechoslovakia	Korea, Dem. P. Rep. of	United Kingdom
Egypt, Arab Rep. of	Korea, Rep. of	U.S.A.
Germany	New Zealand	U.S.S.R.
Greece	Norway	Yugoslavia

The Member Body of the following country expressed disapproval of the Recommendation on technical grounds :

France

The Member Body of the following country disapproved the transformation of ISO/R 1055 into an International Standard :

Spain

Printed in Switzerland

Zinc and zinc alloys — Determination of iron content — Spectrophotometric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a spectrophotometric method for the determination of the iron content of zinc and zinc alloys.

The method is applicable to zinc alloys defined in ISO/R 301, and to die castings made from these alloys, and to zinc containing more than 0,01 % of copper.

It is suitable for the determination of iron contents between 0,01 and 0,08 %.

2 REFERENCES

ISO/R 301, *Zinc alloy ingots.*

ISO 3752, *Zinc alloy ingots — Selection and preparation of samples for chemical analysis.*[1]

3 PRINCIPLE

Spectrophotometric determination of the yellow colour of the sulphosalicylic acid ferric complex formed in an ammoniacal solution after elimination of copper.

4 REAGENTS

During the analysis, use only reagents of analytical reagent grade and only distilled or demineralized water.

4.1 Pure granulated cadmium, free from iron.

4.2 Hydrochloric acid, ρ 1,19 g/ml.

4.3 Hydrochloric acid, ρ 1,19 g/ml, 50 % (*V/V*) (approximately 6 N solution).

4.4 Hydrogen peroxide, 30 % (*m/m*) H_2O_2.

4.5 Sulphosalicylic acid, 400 g/l solution.

4.6 Ammonia solution ρ 0,91 g/ml.

4.7 Iron, standard solution

Weigh 0,250 g of pure iron to the nearest 0,001 g and attack with a few millilitres of hydrochloric acid (4.2). Oxidize with a few drops of hydrogen peroxide (4.4). Decompose the excess hydrogen peroxide by boiling. Cool. Transfer quantitatively to a 1 l volumetric flask. Dilute to the mark and mix. Transfer 100 ml of the solution to a 500 ml volumetric flask. Dilute to the mark and mix.

1 ml of this solution contains 0,050 mg of iron.

5 APPARATUS

Ordinary laboratory apparatus and

5.1 Spectrophotometer, wavelength 425 nm, and **1 cm cells.**[2]

6 SAMPLING

Sampling shall be carried out in accordance with the requirements of ISO 3752.

7 PROCEDURE

7.1 Test portion

Weigh, to the nearest 0,01 g, 10 g of the test sample.

7.2 Blank test

Simultaneously with the actual determination, carry out a blank test using the same quantities of each reagent and following the same procedure.

7.3 Plotting of the calibration curve[3]

7.3.1 Into a series of 100 ml volumetric flasks, introduce 0, 2, 5, 10 and 20 ml respectively of the standard iron solution (4.7). Dilute to about 50 ml.

1) At present at the stage of draft.

2) The dilutions and aliquot parts defined in this International Standard only apply if 1 cm cells are used. It is necessary to apply the appropriate modifications in the case of cells with other dimensions.

3) Valid for 1 cm cells and a range of contents of 0 — 0,1 — 0,25 — 0,5 and 1 mg of iron corresponding to contents of 0 — 0,01 — 0,025 — 0,05 and 0,1 % respectively. It is necessary to apply the appropriate modifications in the case of cells with other dimensions.

7.3.2 Add successively

– 5 ml of sulphosalicylic acid solution (4.5),

– ammonia solution (4.6) until the solution has a yellow colour, then 20 ml in excess.

7.3.3 Cool. Make up the volume to 100 ml with water. Mix.

7.3.4 Measure the absorbance of the solutions against the solution to which no iron has been added in the spectrophotometer (5.1) at a wavelength of 425 nm.

7.4 Determination

7.4.1 Transfer the test portion to a 500 ml conical flask and attack with 100 ml of hydrochloric acid solution (4.3).

7.4.2 Oxidize and complete the solution by adding 1 ml of hydrogen peroxide (4.4).

7.4.3 Decompose the excess of hydrogen peroxide by boiling.

7.4.4 Add about 2 g of cadmium (4.1) and heat gently for 3 min, shaking frequently in order to reduce the copper completely.

7.4.5 Cool. Filter and wash through a rapid filter paper collecting the solution and washings quantitatively in a 250 ml volumetric flask. Dilute to the mark and mix.

7.4.6 Transfer a 25 ml aliquot to a 100 ml volumetric flask.

Add successively

– 5 ml of sulphosalicylic acid solution (4.5),

– ammonia solution (4.6) until the solution has a yellow colour, then 20 ml in excess.

Cool. Dilute to the mark and mix.

7.5 Spectrophotometric measurement

Measure the absorbance of the solution against the blank solution (see 7.2) in the spectrophotometer (5.1) at a wavelength of 425 nm.

8 EXPRESSION OF RESULTS

Determine the iron content by means of the appropriate calibration curve (see 7.3).

9 TEST REPORT

The test report shall mention the method used and the results obtained. It shall also mention all operational details not provided for in this International Standard, or any optional details, as well as any circumstances which could have influenced the results.

The test report shall include all details required for complete identification of the sample.